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(54) **Hot melt jet ink composition**

Heissschmelzende Tintenstrahlzusammensetzung

Composition d'encre thermofusible pour l'impression par jet

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- **Book no. , 1987, 'INDEX OF POLYMER TRADE NAMES', FACHINFORMATIONSZENTRUM CHEMIE GMBH VCH VERLAGSGESELLSCHAFT MBH, WEINHEIM (D)**

EP 0 709 439 B1

Description

[0001] This invention relates to hot melt jet ink compositions. More specifically, this invention is directed to hot melt jet ink compositions especially suitable for printing onto substrates which are stored at low temperatures. The hot melt jet inks provide excellent adhesion, resistance to offset, scratch resistance, and rub resistance at standard refrigerator and freezer temperatures.

[0002] Typically, ink jet printing involves forming characters on a substrate by ejecting ink droplets from a printhead having one or more nozzles. In order to provide a high quality image, hot melt jet ink compositions which are solid at room temperature, but molten at jetting temperatures are used. Unfortunately, such hot melt jet ink compositions, while capable of providing a high quality image at ambient temperatures, are readily abraded or deformed upon handling of a printed substrate which is stored in a low temperature environment, such as in a refrigerator or freezer. In many cases, such low temperature storage causes the ink to become brittle. As a result, the ink may crack or even worse, flake off the printed substrate. In addition, exposure to low temperature may cause the ink to smear or exhibit poor scratch resistance. Typically, a hot melt composition which has excellent adhesion, scratch resistance and flexibility at low temperature may have too much tack and exhibits undesirable offset and pickoff at ambient temperature. Such unintentional transfer of the ink from a freshly printed substrate is particularly troublesome since hot melt jet ink compositions are applied to substrates which are at ambient temperatures, for example typically not lower than at 4°C (40°F), at the time of application.

[0003] The present invention provides a hot melt jet ink composition as set out in Claim 1 of the accompanying claims.

[0004] A process for making such a hot melt jet ink composition as set out in Claim 18 of the accompanying claims.

[0005] Embodiments of the present ink compositions can exhibit excellent adhesion, flexibility, scratch resistance, and improved resistance to offset and rub resistance, even when subjected to low temperatures. They can provide an ink for hot melt jet ink printers that produces print with a sharp image of high resolution on substrates at low temperatures which is resistant to abrasion. They can provide an ink for hot melt jet ink printers that produces print on substrates stored at low temperature which has improved resistance to offset immediately after printing at ambient temperature.

[0006] A hot melt ink jet composition comprising a colouring material, a resin and a vehicle comprising a first component which does not dissolve the colouring material and a second component which has a higher melting point than the first component and can dissolve the colouring material and the first component is disclosed in EP-A-O 565 735. The first component may be a microcrystalline wax and the second component may be Ester Gum H which is a glycerine ester of a hydrogenated resin.

[0007] For a better understanding of the invention reference will now be made, by way of example, to the accompanying drawings.

[0008] Fig. 1 is an FTIR spectrum of an ink composition according to the invention.

[0009] Fig. 2 is an FTIR spectrum of an ink composition according to the invention after 5 days storage at 140°F superimposed on Fig. 1.

[0010] Fig. 3 is an FTIR spectrum of an ink composition according to the invention after 12 days storage at 140°F superimposed on Fig. 1 and Fig. 2.

[0011] Fig. 4 is a DSC endotherm of an ink composition according to the invention.

[0012] Fig. 5 is a DSC endotherm of an ink composition according to the invention after 5 days storage at 140°F superimposed on Fig. 4.

[0013] Fig. 6 is a DSC endotherm of an ink composition according to the invention after 12 days storage at 140°F superimposed on Fig. 4.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0014] Hot melt jet inks according to the invention which are suitable for use in ink jet printers typically have a melt viscosity of at least 10 centipoise, preferably at least 20 centipoise, most preferably in the range from 20 to 25 centipoise, at a temperature of at least 105°C, preferably from 115°C to 140°C, most preferably at 130°C to 140°C. Typically, the hot melt ink according to the present invention is jetted at a temperature of about 135°C.

[0015] The ink compositions of the present invention exhibit good flexibility at temperatures at least as low as -18°C (0°F) to 4°C (40°F), yet can be applied to a substrate at ambient temperature. Surprisingly, the inks of the present invention exhibit excellent adhesion, flexibility, scratch resistance, offset resistance and rub resistance at standard refrigerator temperatures of about 4°C (40°F) and/or freezer temperatures at least as low as -18°C (0°F), yet are not very tacky when applied to a substrate of ambient temperature. Remarkably, these ink compositions exhibit resistance to offset resistance and pickoff at ambient temperature, while remaining flexible at low temperature.

[0016] The essential component of the present invention is a glycerol ester of a hydrogenated rosin which contributes to the overall adhesion and cohesive properties of the ink. Typically, the rosin has a softening point not less than 60°C, preferably less than 100°C, most preferably between 80°C to 88°C, an acid number less than 10, and a molecular weight of 500 to 50,000. Most preferred is a rosin such as Foral 85 available from Hercules Incorporated. The rosin is present in an amount of 15% to 75% by weight, preferably 25% to 55% by

weight, most preferably 30% to 45% by weight of the ink composition.

[0017] The hot melt jet ink of the present invention includes a microcrystalline wax. More preferably the microcrystalline wax remains flexible at low temperatures and has a congealing point of from 54°C (130°F) to 77°C (170°F), most preferably from 66°C (150°F) to 71°C (160°F). A preferred microcrystalline wax is Okerin 103 available from Astor Wax Corp., Doraville, Georgia. The microcrystalline wax is present in an amount 15% to 70% by weight, preferably 25% to 65% by weight, most preferably 35% to 60% by weight of the ink composition.

[0018] The ink composition also includes a polyethylene wax which may increase hardness, improve abrasion resistance, decrease tack, increase offset resistance, and add flexibility. Preferably the polyethylene wax is a homopolymer polyethylene with low density and a low average molecular weight. Most preferably the polyethylene wax has a melting point of 90°C-110°C, a density of 0.85g/cm^3 to 0.95 g/cm^3, and an average molecular weight of about 2,000 to 4,500, preferably 2,500-3,500. The polyethylene wax is present in an amount of 10% to 60% by weight, preferably 15% to 40% by weight, most preferably 15% to 30% by weight of the ink composition. A preferred polyethylene wax is Luwax AL3 available from BASF Aktiengesellschaft in Germany.

[0019] Because hot melt jet ink compositions prepared in accordance with the invention are in a hot molten state during jetting, antioxidants to inhibit thermally induced oxidation may be added to the ink composition. Suitable antioxidants include those conventionally used in the art, for example dibutyl hydroxy toluene compounds and the like. Antioxidant is present in the amount of 0.1% to 5.0% by weight, preferably 0.5% to 3.0% by weight of the ink composition.

[0020] Suitable colouring agents, present in amount of at least 0.1% to 9.0% by weight, preferably 0.5% to 3.0% by weight of the ink composition include pigments and dyes. Any dye or pigment may be chosen provided it is capable of being dispersed in the ink composition and is compatible with the other ink components. Any pigment particles should have a diameter of less than 1 micron. Preferred dyes include Nitrofast Blue 2B (C.I. Solvent Blue 104), Morplus Magenta 36 (C.I. Solvent Red 172), Oracet Yellow GHS, and, for black ink, combinations thereof.

[0021] Hot melt jet ink compositions of the present invention are generally prepared by combining together all the ink ingredients except for the colouring agent and glycerol ester of the hydrogenated rosin, heating the mixture to its melting point, which generally does not exceed 135°C, and slowly stirring until the mixture is homogeneous. The glycerol ester of the hydrogenated rosin is then added to the molten mixture. The colouring agent is subsequently added to this mixture containing the glycerol ester of the hydrogenated rosin while stirring until homogeneously dispersed. The molten mixture is then filtered to remove particles larger than 1 micron in size.

[0022] Specific embodiments of hot melt jet ink compositions in accordance with the present invention will now be described in detail. These examples are intended to be illustrative, and the invention is not limited to the materials set forth in these embodiments. All parts are by weight of the ink composition unless otherwise indicated.

Example 1

[0023]

Ingredient	Parts
Foral 85	39.22
Okerin 103	39.22
Luwax AL3	19.61
Irganox (RTM) 1010	0.98
Nitrofast Blue 2B	0.98

[0024] A hot melt jet ink prepared in accordance with Example 1 resulted in a composition exhibiting excellent flexibility at -18°C (0°F) and excellent resistance to offset after printing at ambient temperature, particularly 10°C (50°F) to 23°C (74°F). Irganox (RTM) 1010 is a derivative of dibutyl hydroxycinnamate available from Ciba-Geigy Corporation.

Example 2

[0025] The low temperature performance of an ink prepared according to Example 1 was determined as follows. A sample of the ink composition (1.5 g) was placed into an aluminium dish having a 2-inch diameter. The ink was melted thereby forming a film in the aluminium dish. The ink containing aluminium dish was stored in a freezer having a temperature of -4°C (25°F) for at least 15 minutes. The aluminium dish was then flexed in the low temperature environment. No ink cracking or brittleness was observed.

Example 3

[0026] The low temperature performance of an ink prepared according to Example 1 applied to some of the typical surfaces used in food packaging was determined as follows. The ink was jetted onto the following substrates:

oriented polypropylene with a polyamide coating
metallized film laminated on paper
ethylene vinyl acetate/polyethylene copolymer coated paper
polyethylene
high slip oriented polypropylene

[0027] The printed substrates were stored in a freezer having a temperature of -18°C (0°F) for at least 15 minutes. The printed substrates were then flexed in the low temperature environment. Hardly any ink flaking was observed for any of the printed substrates.

Example 4

[0028] The thermal stability of an ink composition prepared according to Example 1 was determined as follows. A 10 gram sample of the ink was placed in an aluminium dish. The dish was placed in a forced hot air oven at 140°C for 12 days. A portion of the ink was removed and melted onto a KBr disk and the Fourier Transform Infrared (FTIR) spectrum was recorded. The FTIR spectrum of the sample on day 1 prior to heating at 140°C for 12 days is shown in Figure 1. The FTIR spectrum at day 1 and day 5 is shown in Figure 2. The FTIR spectrum at day 12 superimposed on the spectrum obtained at day 5 and day 1 is shown in Figure 3. As can be seen from Figure 3, no significant shifts in the FTIR spectrum of the hot melt ink composition were observed over 12 days of storage at 140°C.

[0029] In addition, differential scanning calorimetry (DSC) endotherms were obtained using an approximate 5 mg ink sample. Figure 4 is a DSC endotherm for the ink on day 1 before storage at 140°C. Figure 5 shows the DSC endotherm for a 4.9 mg ink sample after 5 days of storage at 140°C superimposed on Figure 4. Figure 6 shows the DSC endotherm measured for a 5.7 mg ink sample after 12 days of storage at 140°C superimposed on Figure 4. As can be seen from Figure 5, no significant changes in the DSC endotherms were observed.

[0030] Finally, the viscosity of the ink composition was determined at 135°C giving the following result:

Day	Viscosity (centipoise)
1	22.4
12	25.9

[0031] These tests indicated ink compositions according to the invention have excellent thermal stability at temperatures as high as 140°C.

[0032] Hot melt jet ink compositions according to the present invention are particularly suited for use in ink jet printers using piezo-electric (drop-on-demand) ink supply systems.

Claims

1. A hot melt jet ink composition for application to a substrate, said composition comprising:

   (a) a glycerol ester of a hydrogenated rosin;
   (b) microcrystalline wax; and
   (c) a colouring agent; characterised in that the composition comprises
   (d) a polyethylene wax.

2. The composition of Claim 1, wherein the rosin component (a) has a softening point of 60-100°C.

3. The composition of Claim 1, wherein the rosin component (a) has a softening point of 60-88°C.

4. The composition of Claim 1, 2 or 3, wherein the rosin component (a) has an acid number less than 10.

5. The composition of Claim 1, 2 or 3, wherein the rosin component (a) has a molecular weight of 500-50,000.

6. The composition of any preceding claim, wherein the rosin component (a) is present in an amount of 15-75% by weight of the hot melt jet ink composition.

7. The composition of Claim 6, wherein the rosin component (a) is present in an amount of 25-55% by weight of the hot melt jet ink composition.

8. The composition of Claim 7, wherein the rosin component (a) is present in an amount of 30-45% by weight of the hot melt jet ink composition.

9. The composition of any preceding claim, wherein the microcrystalline wax is present in amount of 15-70% by weight of the hot melt jet ink composition.

10. The composition of Claim 9, wherein the microcrystalline wax is present in an amount of 25-65% by weight of the hot melt ink jet composition.

11. The composition of Claim 10, wherein the microcrystalline wax is present in an amount of 35-60% by weight of the hot melt ink jet composition.

12. The composition of any preceding claim, wherein the colouring agent is present in an amount of 0.5% to 3.0% by weight of said composition.

13. The composition of any preceding claim, wherein the colouring agent is a dye.

14. The composition of any preceding claim, wherein the colouring agent is a pigment.

15. The composition of any preceding claim, wherein the polyethylene wax has a melting point of 90-110°C, a density of 0.85-0.95 g/cm^3 and an average molecular weight of 2000-4500.

16. The composition of any preceding claim, wherein

the polyethylene wax is present in an amount of 15-40% by weight of the ink composition.

17. The composition of Claim 16, wherein the polyethylene wax is present in amount of 15-30% by weight of the ink composition.

18. A process for making a hot melt ink jet composition as defined in any of Claims 1 to 17, comprising the steps of melting a microcrystalline wax, adding a polyethylene wax to the molten microcrystalline wax, adding to the molten microcrystalline wax a glycerol ester of a hydrogenated rosin, and adding a colouring agent.

19. A process for printing on a substrate which comprises applying imagewise onto the substrate by jet ink printing a composition as claimed in any of Claims 1 to 17.

20. The process of Claim 19, wherein the ink is jetted onto the substrate at a temperature of 115-140°C.

21. The process of Claim 20, wherein the ink is jetted onto the substrate at a temperature of 130-140°C.

22. The process of any of Claims 18 to 21, wherein the substrate has a surface of plastics or coated paper.

23. The process of any of Claims 19 to 22, wherein the substrate is printed using a piezo-electric ink supply system.

24. Use of a composition as claimed in any of Claims 1 to 17 for the printing of a substrate to be stored at about 4.5°C.

25. Use of a composition as claimed in any of Claims 1 to 17 for the printing of a substrate to be stored at about -18°C.

Patentansprüche

1. Eine heißschmelzende Tintenstrahlzusammensetzung zum Auftragen auf ein Substrat, wobei besagte Zusammensetzung

(a) einen Glycerinester eines hydrierten Terpentinharzes;
(b) mikrokristallines Wachs und
(c) ein Farbmittel

umfaßt, dadurch gekennzeichnet, daß die Zusammensetzung

(d) ein Polyethylenwachs

umfaßt.

2. Die Zusammensetzung gemäß Anspruch 1, dadurch gekennzeichnet, daß die Terpentinharz-Komponente (a) einen Erweichungspunkt von 60 bis 100 °C hat.

3. Die Zusammensetzung gemäß Anspruch 1, dadurch gekennzeichnet, daß die Terpentinharz-Komponente (a) einen Erweichungspunkt von 60 bis 88 °C hat.

4. Die Zusammensetzung gemäß Anspruch 1, 2 oder 3, dadurch gekennzeichnet, daß die Terpentinharz-Komponente (a) eine Säurezahl kleiner als 10 hat.

5. Die Zusammensetzung gemäß Anspruch 1, 2 oder 3, dadurch gekennzeichnet, daß die Terpentinharz-Komponente (a) ein Molekulargewicht von 500 bis 50.000 hat.

6. Die Zusammensetzung gemäß jedem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß die Terpentinharz-Komponente (a) in einer Menge von 15 bis 75 Gewichtsprozent der heißschmelzenden Tintenstrahlzusammensetzung vorliegt.

7. Die Zusammensetzung gemäß Anspruch 6, dadurch gekennzeichnet, daß die Terpentinharz-Komponente (a) in einer Menge von 25 bis 55 Gewichtsprozent der heißschmelzenden Tintenstrahlzusammensetzung vorliegt.

8. Die Zusammensetzung gemäß Anspruch 7, dadurch gekennzeichnet, daß die Terpentinharz-Komponente (a) in einer Menge von 30 bis 45 Gewichtsprozent der heißschmelzenden Tintenstrahlzusammensetzung vorliegt.

9. Die Zusammensetzung gemäß jedem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das mikrokristalline Wachs in einer Menge von 15 bis 70 Gewichtsprozent der heißschmelzenden Tintenstrahlzusammensetzung vorliegt.

10. Die Zusammensetzung gemäß Anspruch 9, dadurch gekennzeichnet, daß das mikrokristalline Wachs in einer Menge von 25 bis 65 Gewichtsprozent der heißschmelzenden Tintenstrahlzusammensetzung vorliegt.

11. Die Zusammensetzung gemäß Anspruch 10, dadurch gekennzeichnet, daß das mikrokristalline Wachs in einer Menge von 35 bis 60 Gewichtsprozent der heißschmelzenden Tintenstrahlzusammensetzung vorliegt.

12. Die Zusammensetzung gemäß jedem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Farbmittel in einer Menge von 0,5 Gewichtsprozent bis 3,0 Gewichtsprozent der besagten Zusammensetzung vorliegt.

13. Die Zusammensetzung gemäß jedem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Farbmittel ein Farbstoff ist.

14. Die Zusammensetzung gemäß jedem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Farbmittel ein Pigment ist.

15. Die Zusammensetzung gemäß jedem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Polyethylenwachs einen Schmelzpunkt von 90 bis 110 °C, eine Dichte von 0,85 bis 0,95 g/cm^3 und ein mittleres Molekulargewicht von 2000 bis 4500 hat.

16. Die Zusammensetzung gemäß jedem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Polyethylenwachs in einer Menge von 15 bis 40 Gewichtsprozent der Tintenzusammensetzung vorliegt.

17. Die Zusammensetzung gemäß Anspruch 16, dadurch gekennzeichnet, daß das Polyethylenwachs in einer Menge von 15 bis 30 Gewichtsprozent der Tintenzusammensetzung vorliegt.

18. Ein Verfahren zur Herstellung einer heißschmelzenden Tintenstrahlzusammensetzung wie in jedem der Ansprüche 1 bis 17 definiert, das die Schritte des Schmelzens eines mikrokristallinen Wachses, des Zugebens eines Polyethylenwachses zu dem geschmolzenen mikrokristallinen Wachs, des Zugebens zu dem geschmolzenen mikrokristallinen Wachs eines Glycerinesters eines hydrierten Terpentinharzes und des Zugebens eines Farbmittels umfaßt.

19. Ein Verfahren zum Drucken auf ein Substrat, daß das bildweise Auftragen auf das Substrat durch Tintenstrahldrucken einer Zusammensetzung, wie in jedem der Ansprüche 1 bis 17 beansprucht, umfaßt.

20. Das Verfahren gemäß Anspruch 19, dadurch gekennzeichnet, daß die Tinte auf das Substrat bei einer Temperatur von 115 bis 140 °C aufgespritzt wird.

21. Das Verfahren gemäß Anspruch 20, dadurch gekennzeichnet, daß die Tinte auf das Substrat bei einer Temperatur von 130 bis 140 °C aufgespritzt wird.

22. Das Verfahren gemäß der Ansprüche 18 bis 21, dadurch gekennzeichnet, daß das Substrat eine Oberfläche aus Kunststoffen oder beschichtetem Papier hat.

23. Das Verfahren gemäß der Ansprüche 19 bis 22, dadurch gekennzeichnet, daß das Substrat unter Verwendung eines piezoelektrischen Tintenzufuhrsystems bedruckt wird.

24. Verwendung der Zusammensetzung, wie in jedem der Ansprüche 1 bis 17 beansprucht, für das Bedrucken eines Substrates, um bei etwa 4,5 °C aufbewahrt zu werden.

25. Verwendung der Zusammensetzung, wie in jedem der Ansprüche 1 bis 17 beansprucht, für das Bedrucken eines Substrates, um bei etwa -18 °C aufbewahrt zu werden.

Revendications

1. Composition d'encre thermofusible pour l'impression par jet destinée à être appliquée sur un substrat, ladite composition comprenant :

(a) un glycérolester d'une colophane hydrogénée ;
(b) une cire microcristalline ; et
(c) un agent colorant ; caractérisée en ce que la composition comprend :
(d) une cire de polyéthylène.

2. Composition selon la revendication 1 , dans laquelle le composant de colophane (a) possède un point de ramollissement entre 60 et 100°C.

3. Composition selon la revendication 1 , dans laquelle le composant de colophane (a) possède un point de ramollissement entre 60 et 88°C.

4. Composition selon la revendication 1, 2 ou 3, dans laquelle le composant de colophane (a) présente un indice d'acidité inférieur à 10.

5. Composition selon la revendication 1, 2 ou 3, dans laquelle le composant de colophane (a) a un poids moléculaire de 500 à 50 000.

6. Composition selon l'une quelconque des revendications précédentes, dans laquelle le composant de colophane (a) est présent dans une quantité de 15 à 75% en poids de la composition d'encre thermofusible pour l'impression par jet.

7. Composition selon la revendication 6, dans laquelle le composant de colophane (a) est présent dans

une quantité de 25 à 55% en poids de la composition d'encre thermofusible pour l'impression par jet.

8. Composition selon la revendication 7, dans laquelle le composant de colophane (a) est présent dans une quantité de 30 à 45% en poids de la composition d'encre thermofusible pour l'impression par jet.

9. Composition selon l'une quelconque des revendications précédentes, dans laquelle la cire microcristalline est présente dans une quantité de 15 à 70% en poids de la composition d'encre thermofusible pour l'impression par jet.

10. Composition selon la revendication 9, dans laquelle la cire microcristalline est présente dans une quantité de 25 à 65% en poids de la composition d'encre thermofusible pour l'impression par jet.

11. Composition selon la revendication 10, dans laquelle la cire microcristalline est présente dans une quantité de 35 à 60% en poids de la composition d'encre thermofusible pour l'impression par jet.

12. Composition selon l'une quelconque des revendications précédentes, dans laquelle l'agent colorant est présent dans une quantité de 0,5 % à 3,0% en poids de ladite composition.

13. Composition selon l'une quelconque des revendications précédentes, dans laquelle l'agent colorant est une teinture.

14. Composition selon l'une quelconque des revendications précédentes, dans laquelle l'agent colorant est un pigment.

15. Composition selon l'une quelconque des revendications précédentes, dans laquelle la cire de polyéthylène présente un point de fusion entre 90 et 110°C, une densité de 0,85 à 0,95 g/cm^3 et un poids moléculaire moyen de 2000 à 4500.

16. Composition selon l'une quelconque des revendications précédentes, dans laquelle la cire de polyéthylène est présente dans une quantité de 15 à 40% en poids de la composition d'encre.

17. Composition selon la revendication 16, dans laquelle la cire de polyéthylène est présente dans une quantité de 15 à 30% en poids de la composition d'encre.

18. Procédé de fabrication d'une composition d'encre thermofusible pour l'impression par jet selon l'une quelconque des revendications 1 à 17, comprenant les étapes consistant à faire fondre une cire microcristalline, ajouter une cire de polyéthylène à la cire microcristalline fondue, ajouter à la cire microcristalline fondue un glycérolester d'une colophane hydrogénée, et ajouter un agent colorant.

19. Procédé d'impression sur un substrat qui comprend l'application d'une image sur le substrat par une impression par jet d'encre d'une composition selon l'une quelconque des revendications 1 à 17.

20. Procédé selon la revendication 19, dans lequel l'encre est projetée sur le substrat à une température de 115 à 140°C.

21. Procédé selon la revendication 20, dans lequel l'encre est projetée sur le substrat à une température de 130 à 140°C.

22. Procédé selon l'une quelconque des revendications 18 à 21, dans lequel le substrat comporte une surface de matière plastique ou de papier couché.

23. Procédé selon l'une quelconque des revendications 19 à 22, dans lequel le substrat est imprimé en utilisant un système piézoélectrique d'alimentation en encre.

24. Utilisation d'une composition selon l'une quelconque des revendications 1 à 17 pour l'impression d'un substrat à stocker à 4,5°C environ.

25. Utilisation d'une composition selon l'une quelconque des revendications 1 à 17 pour l'impression d'un substrat à stocker à -18°C environ.

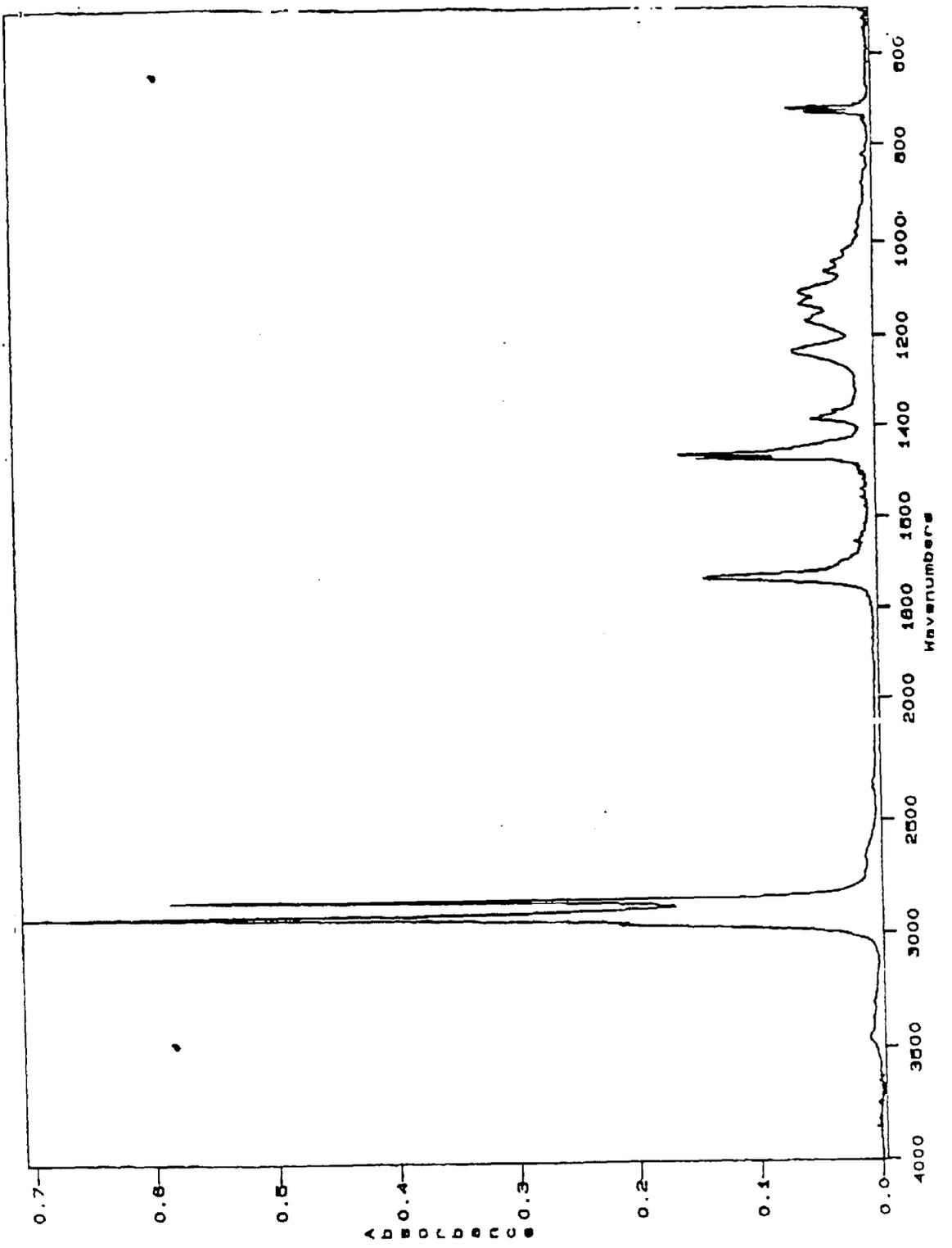
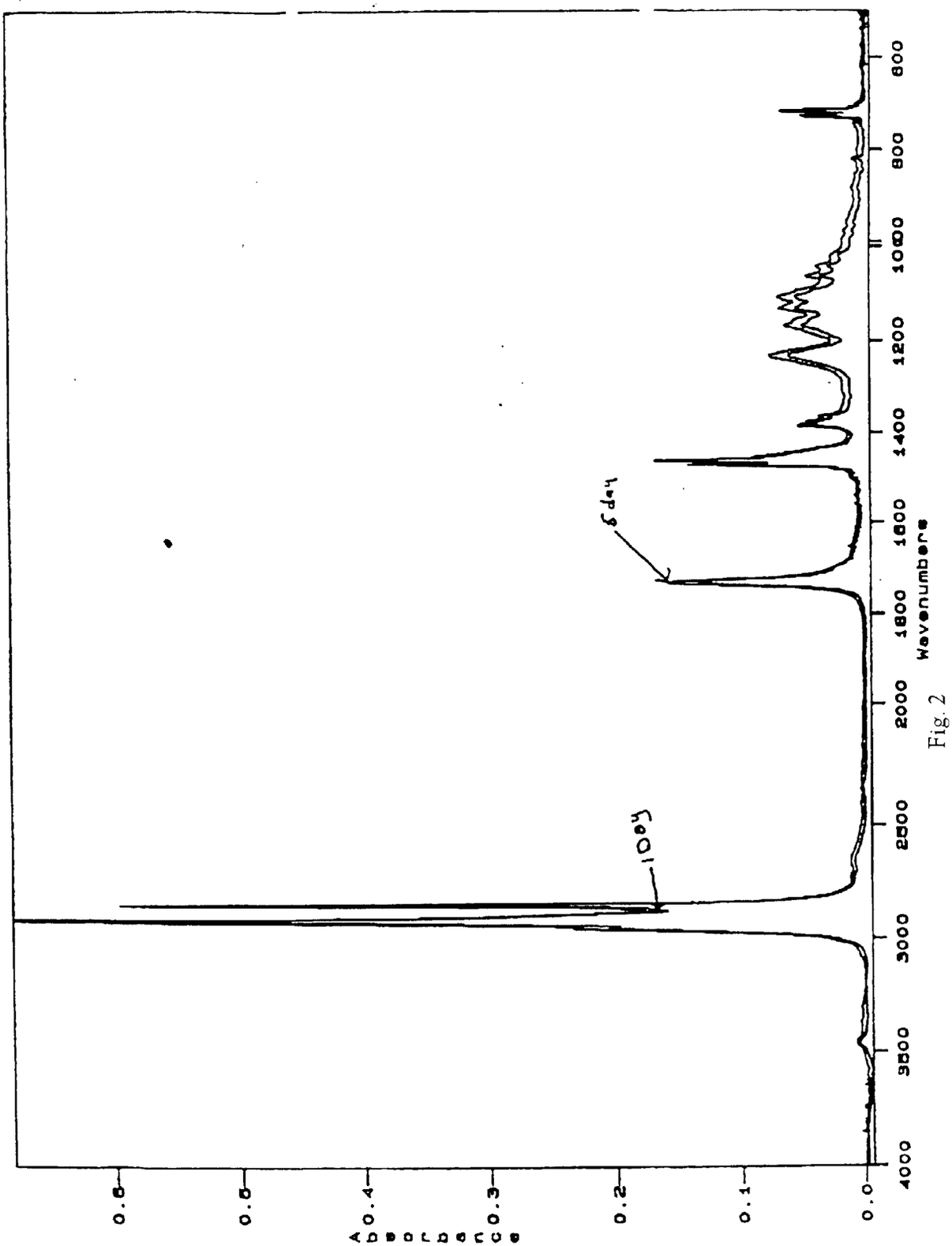

Absorbance
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
4000
3500
3000
2500
2000
1800
1600
1400
1200
1000
800
600
Wavenumbers

Fig. 1


Absorbance
0.0
0.1
0.2
0.3
0.4
0.5
0.6
4000
3500
3000
2500
2000
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1600
1400
1200
1000
800
600
Wavenumbers
1004
1649

Fig. 2

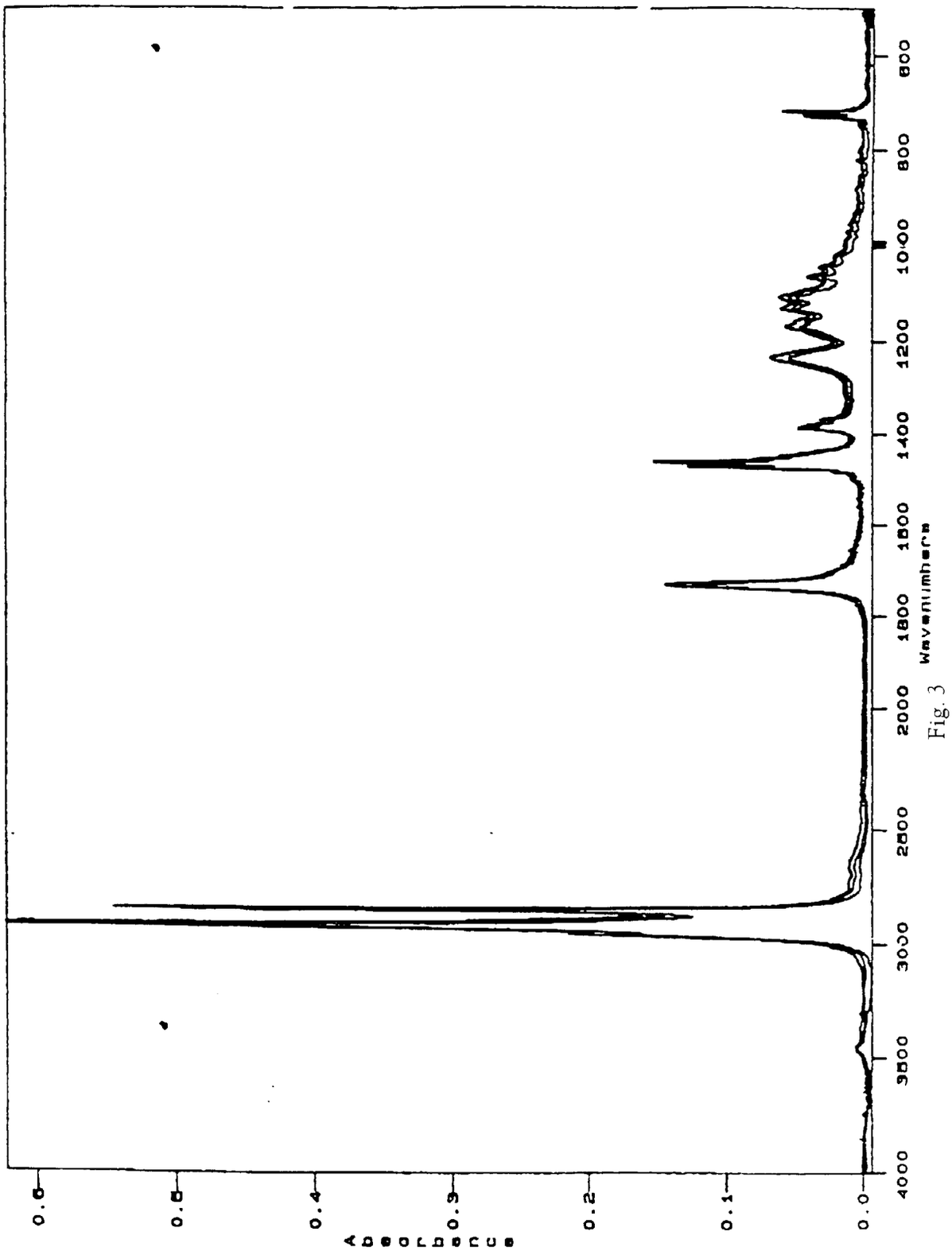
Absorbance
0.0
0.1
0.2
0.3
0.4
0.5
0.6
Wavenumbers
4000
3500
3000
2500
2000
1800
1600
1400
1200
1000
800
600

Fig. 3

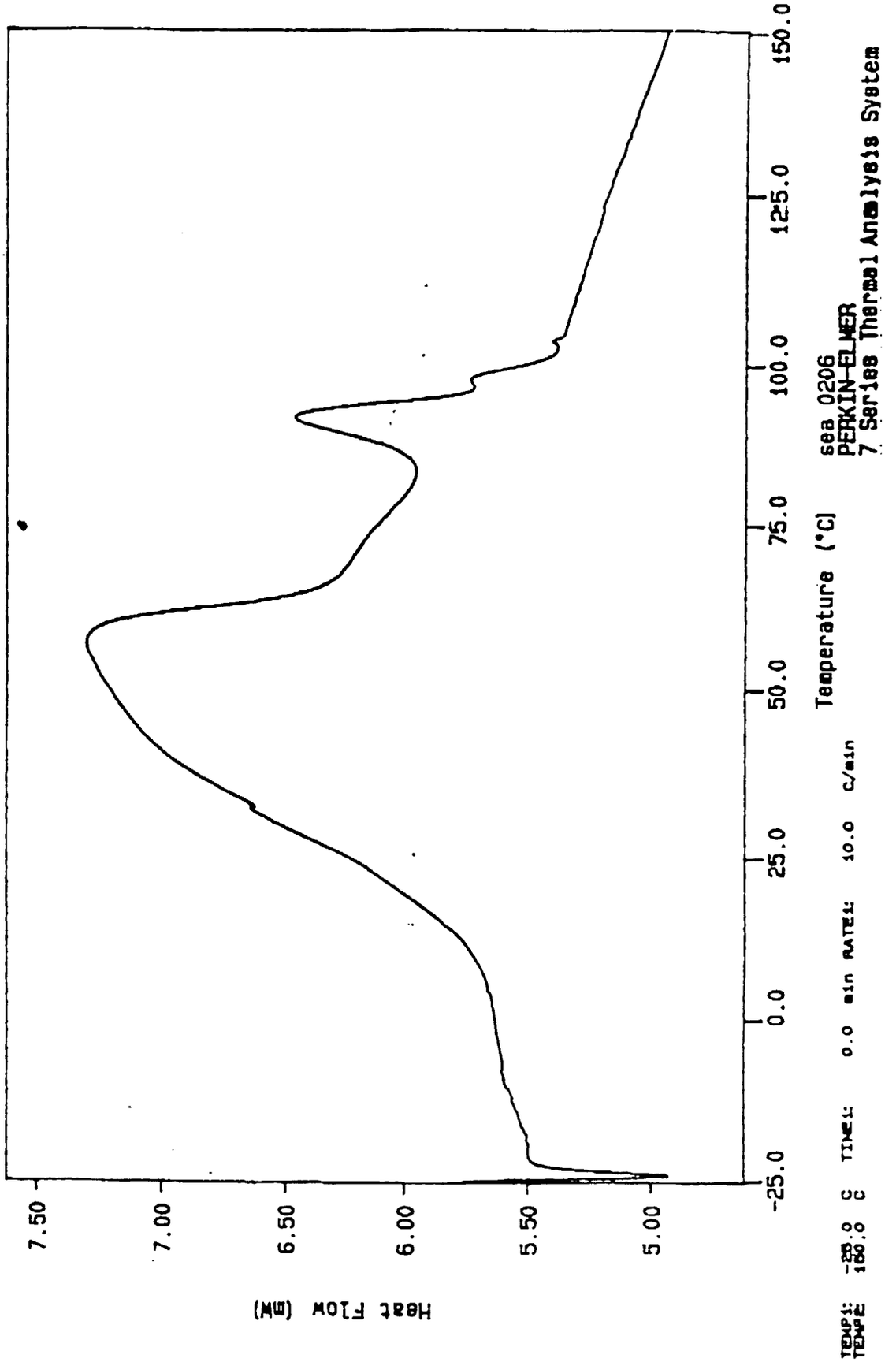
Heat Flow (mW)
7.50
7.00
6.50
6.00
5.50
5.00
-25.0
0.0
25.0
50.0
75.0
100.0
125.0
150.0
Temperature (°C)
sea 0206
PERKIN-ELMER
7 Series Thermal Analysis System
TEMP1: -25.0 C TIME1: 0.0 min RATE1: 10.0 C/min
TEMP2: 150.0 C

Fig. 4

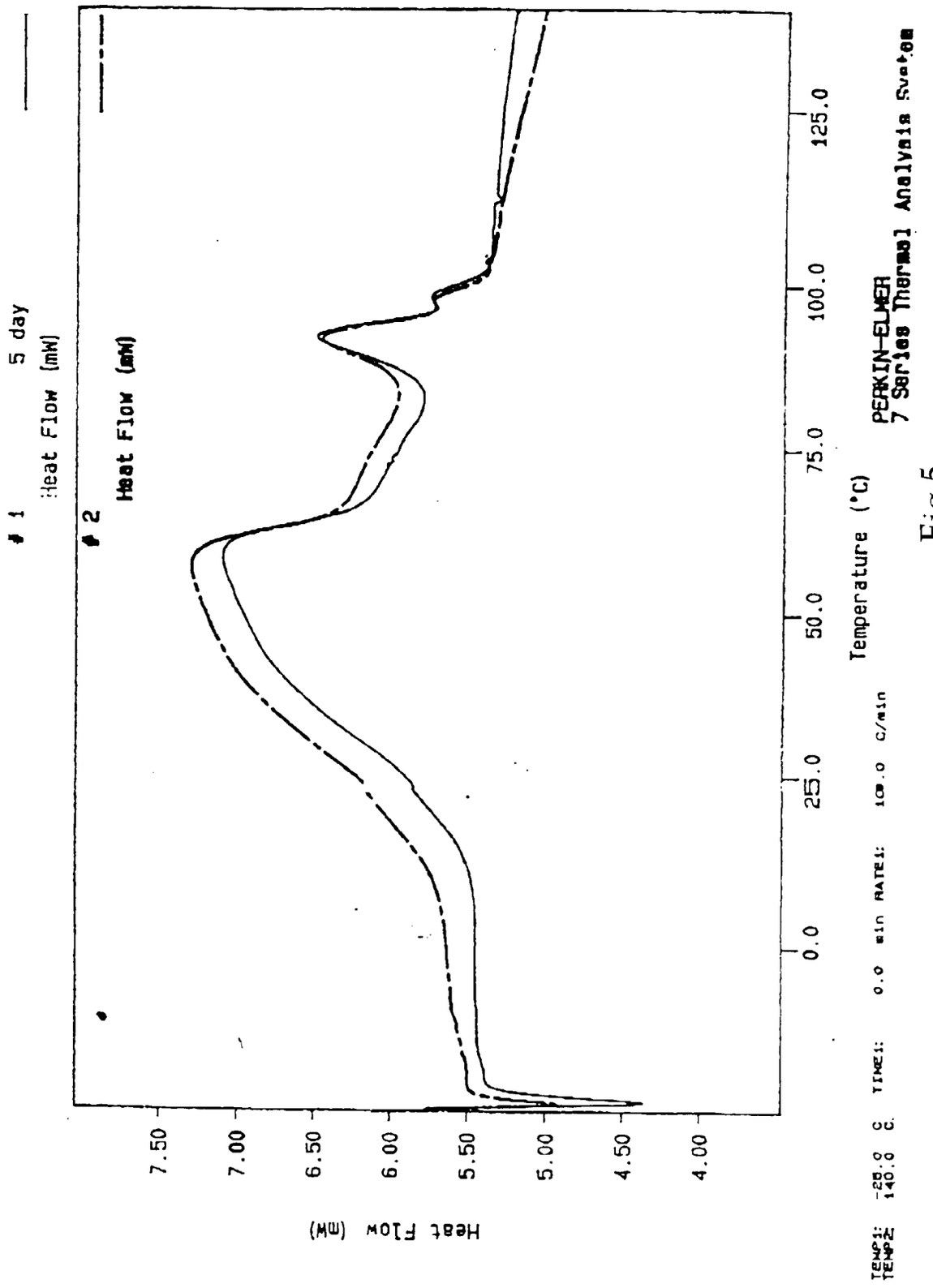
5 day
Heat Flow (mW)
Heat Flow (mW)
Temperature (°C)
PERKIN-ELMER
7 Series Thermal Analysis System
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
0.0
25.0
50.0
75.0
100.0
125.0

Fig.5

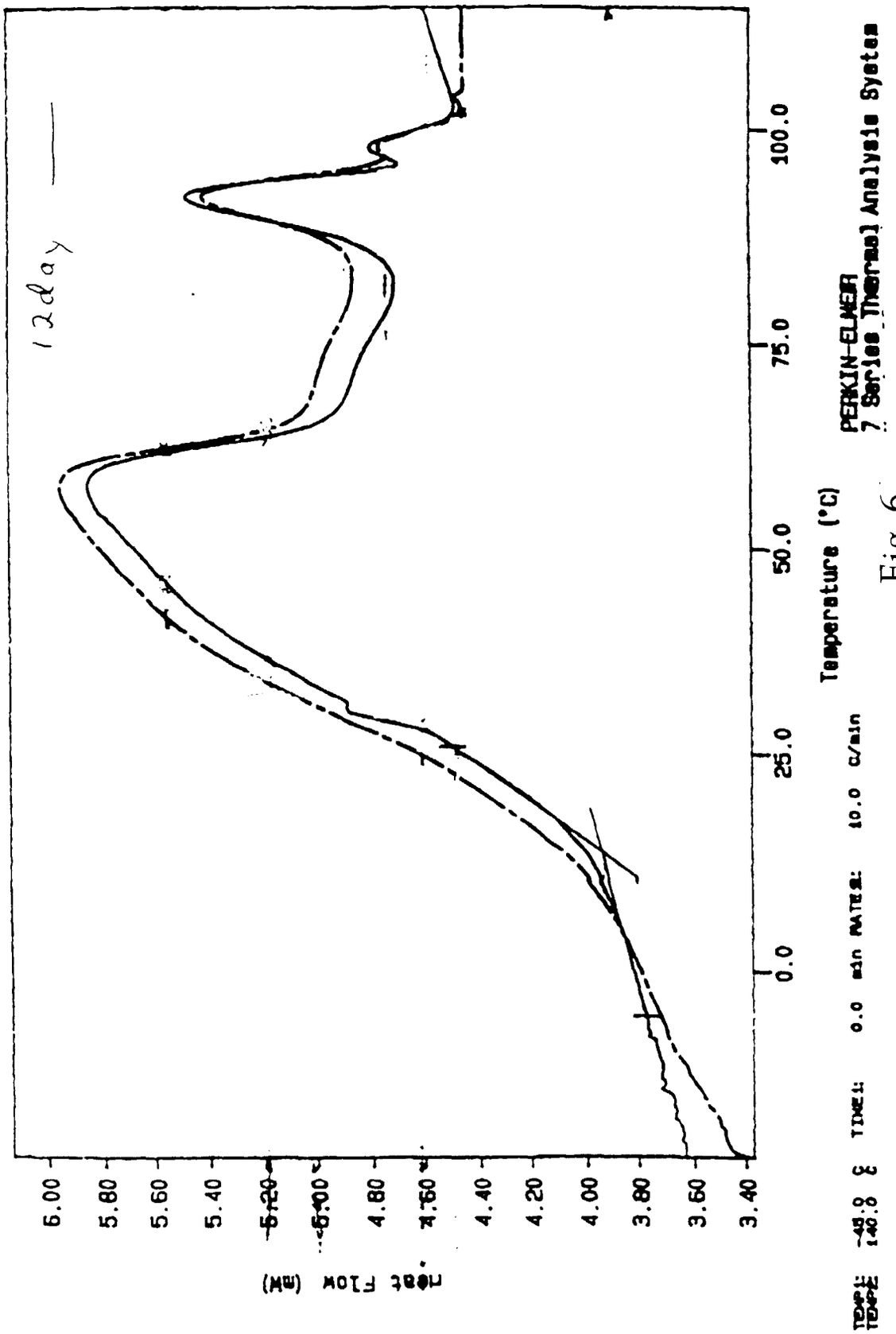
12day
Heat Flow (mW)
6.00
5.80
5.60
5.40
5.20
5.00
4.80
4.60
4.40
4.20
4.00
3.80
3.60
3.40
0.0
25.0
50.0
75.0
100.0
Temperature (°C)
PERKIN-ELMER
7 Series Thermal Analysis System

Fig. 6